Great Ships Initiative

GREAT SHIPS INITIATIVE (GSI)

TECHNICAL SYSTEMS AUDIT CHECKLIST FOR SAMPLES COLLECTED DURING DISCHARGE

Purpose/Scope of Audit:	**GSI Research, Development, Testing, and Evaluation (RDTE) Facility Technical Systems Audit**
Brief Description of Audit:	Audit of sample labeling, collection, transport, and analysis at the GSI RDTE Facility during performance evaluation of the Siemens SiCURE Ballast Water Management System (Trial #6).
Auditee:	GSI scientists
Audit Location:	RDTE Facility (Superior, WI)
Auditors:	Kelsey R. Prihoda, GSI Assistant Quality Assurance Manager
Audit Dates:	Friday, September 25, 2009

SAMPLE BOTTLE LABELING, SAMPLE COLLECTION, AND SAMPLE TRANSPORT TO UWS

SAMPLE TEST ID: 09-SI-6D

Relevant GSI SOPs:

- *GSI/SOP/G/RA/SC/3 – Procedure for Labeling Samples Collected at the GSI Land-Based RDTE Facility (DRAFT)*
- *GSI/SOP/LB/G/O/5 – Procedure for Injecting Organisms and Solids into the GSI Land-Based RDTE Facility*
- *GSI/SOP/LB/RA/SC/3 – Procedure for Algae/Small Protozoa Sample Collection*
- *GSI/SOP/LB/RA/SC/4 – Procedure for Microbial Sample Collection*
- *GSI/SOP/LB/RA/SC/6 – Procedure for Zooplankton Sample Collection*
- *GSI/SOP/LB/RA/SC/3 – Procedure for Collecting Physical/Chemical Data and Samples at the GSI Land-Based RDTE Facility (DRAFT)*

➢ **Drain Scenario:** ☐ **Drain Treatment Tank First** ☑ **Drain Control Tank First**

➢ **Time Discharge Treatment Tank Started:** 11:05 am **Time Discharge Treatment Tank Completed:** 11:59 am

➢ **Time Discharge Control Tank Started:** 8:46 am **Time Discharge Control Tank Completed:** 9:40 am

Sample Collection Type (Code)	Sample Port/Point	Tub Number	Sample Type (Collected By)	Labeled Correctly & In Crate? Y	Labeled Correctly & In Crate? N	Collected Following SOPs? Y	Collected Following SOPs? N	Transported Back to UWS? Y	Transported Back to UWS? N
Control Tub (C)	SP9-C	1	Phytoplankton	✓		✓ 9:41 am			
			Zooplankton	✓		✓ Recd 10:07			
			Microbe Rep. 1	✓		✓ 9:42		✓	
			Microbe Rep. 2	✓		✓ 9:42		✓	
			Microbe Rep. 3	✓		✓ 9:42		✓	
			TRC and TRO	✓		✓ 9:47			

Tom marker to transport ↓

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Sample Collection Type (Code)	Sample Port/Point	Tub Number	Sample Type (Collected By)	Labeled Correctly & In Crate?		Collected Following SOPs?		Transported Back to UWS?		
				Y	N	Y	N	Y	N	
			Disinfection Byproducts (2 L)	✓		✓ 9:43 am		✓		Tom marked to Transport.
Treatment Tub #4 (T)	SP9-C	4	Phytoplankton	✓		✓ 12:00 pm				
			Zooplankton	✓		✓				
			Microbe	✓		✓ 12:01 pm		✓		
			TRC and TRO	✓		✓ 12:01 pm				
Treatment Tub #5 (T)	SP9-B	5	Phytoplankton	✓		✓ 12:02 pm				
			Zooplankton	✓		12:35 pm Rec'd				
			Microbe	✓		✓ 12:02 pm		✓		Tom marked to transport.
			TRC and TRO	✓		✓ 12:02 pm				
Treatment Tube #6 (T)	SP9-A	6	Phytoplankton	✓		✓ 12:04 pm				
			Microbe	✓		✓ 12:04 pm		✓		
			TRC and TRO	✓		✓ 12:04 pm				
			TRC and TRO Duplicate	✓		✓ 12:05 pm				
			Whole Effluent (~38 L)	✓		✓ 12:05 pm		✓		Christine Polking normal to transport
			Disinfection Byproducts (2 L)	✓		✓ 12:05 pm		✓		
Treatment Tank 2	Mid-Depth		TRC Monitoring – Day 5	✓		✓				

Treatment Tank 1 Mid-Depth TRC Monitoring – Day 4 ✓

ADDITIONAL QUESTIONS/COMMENTS FROM SAMPLE COLLECTION:

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SAMPLE ANALYSIS

SAMPLE TEST ID: 09-SI-6D

QUALITY SYSTEM DOCUMENTATION Please see 09 SI 6 Fill TSK for answers to questions #1-#10.

AUDIT QUESTIONS	RESPONSE			COMMENTS
	Y	N	NA	
1. Is there an approved Quality Assurance Project Plan for the overall project and has it been reviewed by all appropriate personnel?				
2. Is a copy of the current approved QA Project Plan maintained near laboratory work station areas?				
3. Is the implementation of the project in accordance with the QA Project Plan?				
4. Are there deviations from the QA Project Plan? Explain.				
5. Do any deviations from the QA Project Plan affect data quality?				
6. Are sample handling and storage procedures in accordance with the QA Project Plan?				
7. Are written and approved current standard operating procedures (SOPs) used in the project? If so, list them and note whether they are maintained near laboratory work station areas?				
8. Are data/observations appropriately recorded in laboratory notebooks/forms according to the QA Project Plan (i.e., entries in ink, dated, initialed, corrections done properly)? Are data contained in bound, well-labeled notebooks or three-ring binders?				
9. Do supervisory and/or QA personnel inspect laboratory notebooks/forms on a regular basis and initial notebook after review?				
10. Are paper records written in indelible ink?				
Additional Questions or Comments:				

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CHEMISTRY

Relevant GSI SOPs:

- *GSI/SOP/BS/RA/C/2 – Procedure for Determining Total Residual Oxidants (TRO) in Water*
- *GSI/SOP/BS/RA/C/3 – Procedures for Measuring Organic Carbon in Aqueous Samples*
- *GSI/SOP/BS/RA/C/6 – Procedure for Analyzing Total Residual Chlorine (TRC) Concentrations in Water*
- *GSI/SOP/BS/RA/C/8 – Procedure for Analyzing Total Suspended Solids (TSS)*

Chemical analyses not observed during Trial 6 Discharge.

AUDIT QUESTIONS	RESPONSE Y	N	NA	COMMENTS
1. Describe the analytical instrumentation. List the brand and model number for each instrument.				
2. Are calibration and maintenance logs kept for the instrumentation (e.g., balances and other equipment)?				
3. Review the maintenance and operational records for the equipment. Based on your findings, do all instruments/equipment appear to be in good operating condition?				
4. Are the manufacturer's operating manuals readily available to the instrumentation operators?				
5. Describe the routine calibration procedure.				
6. Does the calibration documentation show that the calibration procedures are being followed?				
7. Do the calibration standards have the appropriate levels (i.e., bracket the samples to be measured)?				
8. What is the instrumentation calibration error according to the calibration documentation?				
9. Are duplicate samples collected and analyses conducted on at least 10% of the physical/chemical samples?				
10. Are reagent blank samples analyzed with each set of samples?				
11. Are a minimum of three and preferably more standards required for standard curves?				
12. When applicable, do routine procedures that require standard curves bracket concentrations?				
13. When applicable, have analytical method detection limits been established and clearly documented?				

Additional Questions or Comments:

Treatment Tank 2 = 70 µg/mL OR 0.070 mg/L

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MICROBIOLOGY – Did not observe microbial analyses done post-discharge.
Relevant GSI SOPs: Reviewed datasheets 23 Oct. 2009 MP.

- *GSI/SOP/BS/RA/MA/1 – Procedure for Quantifying Heterotrophic Plate Counts (HPCs) using IDEXX's SimPlate® for HPC Method*
- *GSI/SOP/BS/RA/MA/3 - Procedure for the Detection and Enumeration of Enterococcus using Enterolert™*
- *GSI/SOP/BS/RA/MA/4 – Procedure for the Detection and Enumeration of Total Coliforms and E. coli using IDEXX's Colilert®*

AUDIT QUESTIONS	RESPONSE Y	N	NA	COMMENTS
1. Are duplicate sample analyses conducted on at least 10% of the microbiology samples?	✓			All control EC, TC, + ENT samples done in DUP. 2 HPC + VC DUPs. EC,
2. Are at least 10% of the samples counted by a second qualified individual (i.e., QA count)?		✓		No ENT, TC QA count. No HPC QA count. No VC QA count.
3. Are ~~reagent~~ blank samples analyzed with each set of samples? (RE) MP 10-23-09	✓			
4. When applicable, have analytical method detection limits been established and clearly documented?	✓			

Additional Questions or Comments: Collected control discharge samples at 9:42am. Collected treatment discharge samples at 12:01-12:04pm. Time of sample receipt in laboratory not recorded. Control sample analysis completed by 12:00pm. Treatment sample analysis completed by 2:05pm. E.coli/TC Quantitrays incubated in incubator set at 36°C? No incubator checks done for a 35°C incubator.
Data entry proofed by TJJ 29 Sept. 09. Calculations verified for 10% samples by MP 23 Oct 09.

PHYTOPLANKTON – Observed counting/analysis of control sample (09SI6DSP9-1C)
Relevant GSI SOPs: Reviewed datasheets 19 October 2009. MP.

- *GSI/SOP/LB/RA/SA/1 – Procedure for Algae/Small Protozoan Sample Analysis*

AUDIT QUESTIONS	RESPONSE Y	N	NA	COMMENTS
1. Were all data, observations, and comments appropriately recorded on the "Ballast Water Plankton Count Sheet"?		✓		Control sample data was recorded using pencil. Time of sample analysis was not recorded.
2. Was sample assessment conducted within ~1-1.5 hours after sample collection?	✓			Control: ~9:45am – ~10:30am
3. Were at least 10% of the samples counted by a second analyst (i.e., QA count)?				No QA on Trial 6 discharge. Was there QA on Trial 6 fill? yes: 09SI6FSP3-4 PT.

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Additional Questions or Comments:
It is recommended that whenever possible additional sample volume be concentrated down for analysis in order to increase representativeness and accuracy of density data, within the 1.5 hour time frame. If additional volume can't be concentrated, the entire slide could be counted as opposed to 1-3 transects.

Control = 125 live cells/mL
Treatment = 6 live cells/mL

Control discharge sample mislabeled not a composite of Tubs 1, 2, + 3. A 1L sample from Tub 1 was used as control. Treatment drain datasheet very difficult to read.

ZOOPLANKTON

Relevant GSI SOPs: Reviewed datasheets 20 Oct. 2009. MB

- *GSI/SOP/BS/RA/C/2 – Procedure for Zooplankton Sample Analysis (DRAFT)*

AUDIT QUESTIONS	RESPONSE Y	N	NA	COMMENTS
1. Were all data, observations, and comments appropriately recorded on the "Zooplankton Identification Worksheet"?	✓			Tub 4: 2:11 – 2:40
2. Was sample assessment conducted within ~2 hours after sample collection?	✓			Control Rec'd 10:07 – 11:20 Tub 5: 12:33 – 1:19 Tub 6: 1:25 – 2:04
3. Were at least 10% of the samples counted by a second analyst (i.e., QA count)?	✓			Control sample QA count.

Additional Questions or Comments:
QA count was done on control sample. Many rotifers died between QA count done by HLS at 10:35 am and raw count done by MB at 11:10. This is most likely due to crowding on the slide and the length of time between the two counts. It is recommended that for samples having a QA count, limit organisms per slide to 100 or less, and do a raw count of 3-4 subsamples.

WHOLE EFFLUENT TOXICITY (WET)/COLD WATER BIOASSAY (CWB) TESTING – Set up of Whole Effluent Toxicity Test for Trial 6 was not observed. Reviewed data sheets 07 Oct. 2009.

Relevant GSI SOPs:

- *GSI/SOP/BS/RA/RT/6 – Procedure for Assessing Chronic Residual Toxicity of a Ballast Treatment System to Ceriodaphnia dubia*
- *GSI/SOP/BS/RA/RT/7 – Procedure for Assessing Chronic Residual Toxicity of a Ballast Treatment System to the Fathead Minnow (Pimephales promelas)*
- *GSI/SOP/BS/RA/RT/8 – Procedure for Assessing Chronic Residual Toxicity of a Ballast Water Treatment System to the Green Alga (Selenastrum capricornutum) (DRAFT)*

AUDIT QUESTIONS	RESPONSE Y	N	NA	COMMENTS
1. Were all data, observations, and comments appropriately recorded on pre-printed data sheets	✓			

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and/or laboratory notebooks?				
2. Were all relevant standard operating procedures followed (see above)?				*SOP deviations
3. Was an organism QA count done on at least 10% of the test chambers by a second, qualified analyst?	① ✓			~~② cont~~

Additional Questions or Comments:

*C. dubia were added to test chambers on D0 at 1:35-2:07pm, D1 renewal started at 8:22 am ∴ orgs. were only exposed for ~19 hours.

-Reps. 4, 5, 7, + 9 in the dilution water control never produced young - they may have been males but this was never confirmed. EPA methods state "the occurrence of males in a healthy... culture is rare!" The occurrence of males usually is indicitive of organisms experiencing stress within the culture. C. dubia WET test is not acceptable.

①RE KRL 9-25-09 ②RE KRL 10-7-09

*P. promelas were added to test chambers on D0 at 2:10-2:36 pm, D1 renewal started at 9:15 am ∴ orgs. were only exposed for ~19 hours. Forgot to feed P. promelas 2nd time on Day 1.

Additional Questions and Comments on Technical Systems Audit:

- There were 15 larvae in 6.25%-4 on D0 and D1, then on D2 there were 16.

*Initial cell density for S. capricornutum was 213,333. This is 21 times higher than recommended by EPA WET methods, or directed in the GSI SOP.

- pH in reference control (S. capricornutum) dropped to 3.81 by Test Day 2 and remained significantly lower than all other treatments throughout the study duration.

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